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Report to the

Air Force Office of Scientific Research

on

Time Dependence in Fracture Mechanics

of Adhesive Joints and Composites

by

W.G. Knauss

California Institute of Technology

September 1982

Annual

AFOSR Grant No. AFOSR-81-0127

1. INTRODUCTION

The research program covered in this report is concerned with understanding the time dependent effects involved in the failure of polymer based adhesive joints and in advanced composites. The emphasis is placed on:

a. Examining the effect of time dependent processes that influence failure in bonded structures and in composites

b. Assessing the validity of currently used analytical techniques in predicting failure of adhesive bonds

c. Examining means of accelerated testing which would allow one to make long-term failure predictions on the basis of the relatively short-term tests, under both monotonic and fatigue-type loading.

These basic research objectives have been pursued in several specific problem areas which we describe in the following along with a summary of reporting and coupling activity.

2. COUPLING ACTIVITIES AND REPORTS

During this reporting period, several publications resulted from previous and on-going research.

A graduate student, Luc Heymans, whose studies and research had been supported by this grant (besides support from the DOE) completed his Ph.D. work with a thesis entitled, "An Engineering Analysis of Polymer Film Adhesion to Rigid Substrates."

This work addressed two technical problems, one of which relates equally well to composite as to adhesive technology, and corresponds to points a) and b) in the Introduction; the second problem deals primarily with that of temperature accelerated testing of soft adhesive systems (point c in the Introduction).

The first of these problems is concerned with the residual stresses generated in multiphase systems, such as fiber composites, laminations in bonded joints, or polymer films deposited on rigid substrates (projective polymer film). The problem revolves around residual stresses generated in a polymer that has been cooled through its glass transition temperature - a problem of paramount importance in both composites and in bonding technology. The more specific question asked regards the precision with which such predictions can be accomplished. Thus the study involves both precision computations (exact application of today's knowledge in the field) and precise measurements involving optical interferometry. The result of this study - delineated in the attached thesis and presently being written up for publication - establishes that the limiting factor in that precision is primarily one's ability to determine the rheological parameters of the polymer - be that as a result of inadequate measurement techniques or because of the intrinsic variability of the material. In the present case the latter limitation dominated by far - even though a polymer-standard of sorts had been used. The results showed deviation between computation and measurements on the order of 25%. It is not expected that in a typical industrial engineering environment such "precision" is achieved.

The second problem concerns questions that arise in temperature induced accelerated testing when adhesion phenomena play a role (fuel tank sealants, solid propellant rocket liner adhesion, steel wire and rubber adhesion in tires, gas masks, potting and encapsulation of electronic component in space applications). We find that in this application it may be necessary to account for the energy stored in the polymer due to thermo-differential stressing in order to properly evaluate the interface failure characteristics. This fact is normally disregarded in evaluating the adhesion energy, but from our studies it appears that this neglect is valid only for high adhesive energies.

The first of these problems is in the process of being written up for publication under the title "Temperature Induced Residual Stresses in Glossy Resins. Comparison of Experiment and Theory." The second portion is slated for publication next.

Two previous reports have appeared as publications in the *Experimental Mechanics*, namely,

> "Crack Propagation at Material Interfaces: I. Experimental Technique to Determine Crack Profiles," Vol. 22, No. 7, pp. 262-269, July 1982,

> "Crack Propagation at Material Interfaces: II. Experiments on Mode Interaction," Vol. 22, No. 10, pp. 383-391, October 1982.

In addition to these written accounts the principal investigator participated in a symposium of the Americal Chemical Society (Kansas City, Missouri, Sept. 1982) by presenting a paper Entitled "Interfacial Crack Growth and its Relation to

Crack Front Profiles." This contribution is half completed in written form.

Another presentation on non-linear constitutive behavior of fractured solids (composite propellant rocket fuels) which is related to adhesion problems, was given at the winter meeting of the ASME (Phoenix, Arizona, November 1982) as well as at an NSF workshop on "Damage and Fracture" in Atlanta (Stone Mountain). The title of that paper is "Non-Linear Viscoelastic Material Behavior Including Void and Tear Generation."

3. INTERFACIAL CRACK PROFILOMETRY

In the earlier studies of crack flank displacements of an interface crack (see the paper mentioned in Section 2, "Crack Propagation of Material Interfaces: II. Experiments on Mode Interaction") a crack propagation criterion based on the relative crack flank displacements was developed. The experimental investigation valid up to that point was based on experimentally forcing either bond-normal or bond-parallel displacements of the adherends. Thus a true loading was not yet attempted. That direct interaction was to be the result of the present effort. Accordingly experiments were continued - with a new student - to augment the earlier results with direct mode-interaction measurements.[1]

The experiment depends on a microcomputer-feedback system (see the paper mentioned in Section 2, "Crack Propagation of Material Interfaces: I. Experimental Technique to Determine Crack Profiles") to control the applied displacements. As the time approached where the experimental set-up was ready to commence the measurements, the microprocessor failed by terminating experiments at random times after test start.

We have tried to have the microprocessor repaired through the store of purchase. It has been shipped to the manufacturer who returned it unrepaired with the comment that it is so outdated that circuit schematics are not even available. (The microprocessor was purchased 3/29/78, a life of only 4.5 years!)

We have lost much time with this problem and are now trying to repair the unit in house, but so far with no success.

1. Specifically the goal was to make crack propagation measurements under simultaneous bond-normal and bond-parallel loading, with the ratio between these two types of displacements being varied.

4. A NEW "CRITERION" FOR CRACK STABILITY

Our approach to the failure of bonded joints is via the fracture mechanics discipline. In the event that one deals with a rather ductile adhesive (high strain capability) it is appropriate to consider fracture as the failure of the adhesive averaged across the bond line (cf. Figure 1). In that event the adhesive at the "crack tip" stretches and undergoes deformations that involve non-linear stress-strain behavior. Let this non-linear stress strain behavior allow for unloading in the sense that for increasing strains the stress that can be sustained decreases (as shown in Figure 2 for different types of materials). As one increases the loading the material of the crack tip will strain causing progressive unloading after some maximum stress level has been achieved locally. However, it does not follow that unstable crack growth occurs as soon as local unloading of the crack tip stresses occur; rather stability is governed by the second variation of the system potential energy with respect to the crack tip position.

The solution of this problem contains two phases: The first deals with obtaining the equilibrium solution for the problem with the non-linear cohesive forces at the crack tip. This problem requires the solution of a non-linear integral equation, which is solved by iteration.

The second phase deals with the determination of the load at which the system potential energy has a vanishing second derivative with respect to the crack extension.

Certain aspects of the first phase (two dimensional) have been resolved, in that the iteration scheme for the solution of the non-linear integral equation is functional, including the determination of the initialization conditions to effect convergence of the iteration scheme. As an example of those preliminary comutations we show in Figure 3 a plot of the crack tip displacement and cohesive zone size α for the geometry and (uniaxial) crack tip cohesive stress-strain law shown inset in that figure. The crack tip displacement is the opening displacement at the trailing end of the cohesive zone.

We note that the maximal stress which can be carried occurs when $dv/d\sigma$ or $d\alpha/\alpha\sigma$ becomes unbounded. That point corresponds to the moment when the cohesive force at the trailing edge of the craze reaches the maximal strain and zero cohesive force value. Of course, in the present formulation any solution for the displacements V or α/c is not valid past that point because at that moment in the loading history the crack size would change, so that a new, though geometrically close, geometry would result. In order to assess whether that change in crack size occurs in a stable or unstable manner it is necessary to "expand the problem about this point," and consider the energy balance as the crack tip moves a small amount. That energy balance, and thus the stability criterion, will depend on the rate of unloading (rate with respect to crack extension) at the trailing edge of the cohesive zone as compared with the rate of loading of the tip of the cohesive zone and its advance. This balance of work and thus the stability criterion depends on the derivative of the stress-strain behavior for the cohesive material, but the details are to be worked out in the future.

Although these stability considerations were initiated with the failure of *bonded* structures in mind, identical consideration apply to the fracture of homogeneous solids. In the event that a very slender plastic zone (Dugdale zone) develops, as, for example, in aircraft skins or in some rigid polymers, the computations outlined above are directly applicable.

Accordingly, calculations are being performed at present for assessing the effect which crazed material has on the development of crack opening profiles.

To this end we are at present using the measured (non-linear) stress-strain properties of PMMA crazes as a cohesive model material without making the usual assumption of "small scale yielding." Thus the *development* of crazes can be assessed, as well as - later on - their development into cracks.

5. FATIGUE OF ADHESIVES AND COMPOSITE MATRIX MATERIALS

One of the structurally most important load histories is cyclic in nature and leads to fatigue. In metallic bodies fatigue is usually "explained" in terms of a da/dn-vs-K cure. The question of how this behavior of fatigue crack growth depends on the properties of the material (yield, microstructure, etc.) is usually answered by generating parametric da/dn-vs-K curves.

In polymers the fatigue behavior depends on cycle frequency and temperature much more sensitively than in the structural metals. Therefore it is important to establish under which conditions the conventional approach to fatigue via da/dn-vs-K curves breaks down. For example, at temperatures close to the glass transition which are presently allowed in aircraft design, the damping characteristics of polymers (bonding agents, matrix materials in composites) change and the dissipation at the tip of a crack will affect its propagation rate. Similarly, moisture affects the crack growth rate through changes of the material behavior at the crack tip.

Rather than perform test sequences that assess the effect of each of these parameters the fatigue behavior for any one material it is important to understand from a more fundamental point of view how the intrinsic material properties affect the crack propagation rate under varied conditions. Such understanding is essential for estimating service life of polymer-based designs.

These considerations are important in several flight-structural problems: Besides aircraft structural bonding and failure of composites, these problems surface also in connection with liner and insulation adhesion in solid propellant rockets as in the failure of composite propellants as well as in the failure of fuel tank sealants.

One could argue that the study of fatigue fracture in a pure polymer is of little relevance to the fracture of bonded joints and composite materials because the former does not simulate the latter geometrically: Current aircraft adhesives are really a composite through the use of a scrim layer and in fiber composites the fracture process is strongly influenced by the presence of the fibers. While that is true one must remember that the fracture process is governed by the break-up of the matrix - or fiber matrix interface - and thus the *failure rate is governed by the rate of fracture development in the polymer itself*. Therefore, if loading conditions and thermal/humidity environment influence the rate of fatigue growth in homogeneous polymers, the same effects will be present in these geometrically more complicated structures. It is on the basis of this rationale that we explore the fatigue in the form of crack propagation behavior for a homogeneous polymer.

This we do - on a rather small scale - by examining the crack propagation rate of an elastomer in the glass transition range as a function of frequency. Initially tremendous data scatter clouded the data analysis so that a careful effort needed to be made to reduce that scatter. This is being accomplished now by recording data on video tape and to analyze the results with slow - step wise - play back.

Both the location of the crack tip as a function of cycles and the corresponding crack tip stress field (stress intensity factor) are recorded. The latter is accomplished via the optical method of caustics, rather than by computation or photoelastic means.

6. WORK DURING THE REMAINDER OF THE GRANT

There are three areas where work will be concentrated.

1) First, we shall continue to try repairing the microprocessor. We have considered buying a new one. However, though the cost would be bearable, it turns out that the same one is no longer available and a new, updated system would require the development of the software for the servo-function. In the light of our previous experience with that development this alternative does not seem viable. Thus the cost according to the progress from this effort is only the cost of the repair although that requires more waiting time than lower cost.

2) Second, we continue to exploit our capability with the computations of non-linear material response between the flanks of adherends (crack faces). Our immediate goal is to examine the development of a crack from a craze. The next goal is to show that the *toughening of matrix materials* achieved by the addition of small rubber particles results from the highly non-linear stress strain behavior of the rubber particle that acts like a strong (non-linear) spring between the faces of a craze crack. We presume that it is this strong cohesive force that prevents the craze crack from spreading and causing material rupture. At the same time we begin to explore the formulation of the energy considerations that control crack stability.

3) Finally we shall complete our initial investigation into fatigue in a viscoelastic material. We expect that this preliminary investigation will delineate the degree and range of deviation of measurements made in the standard description of fatigue when viscoelastic effects must be considered.

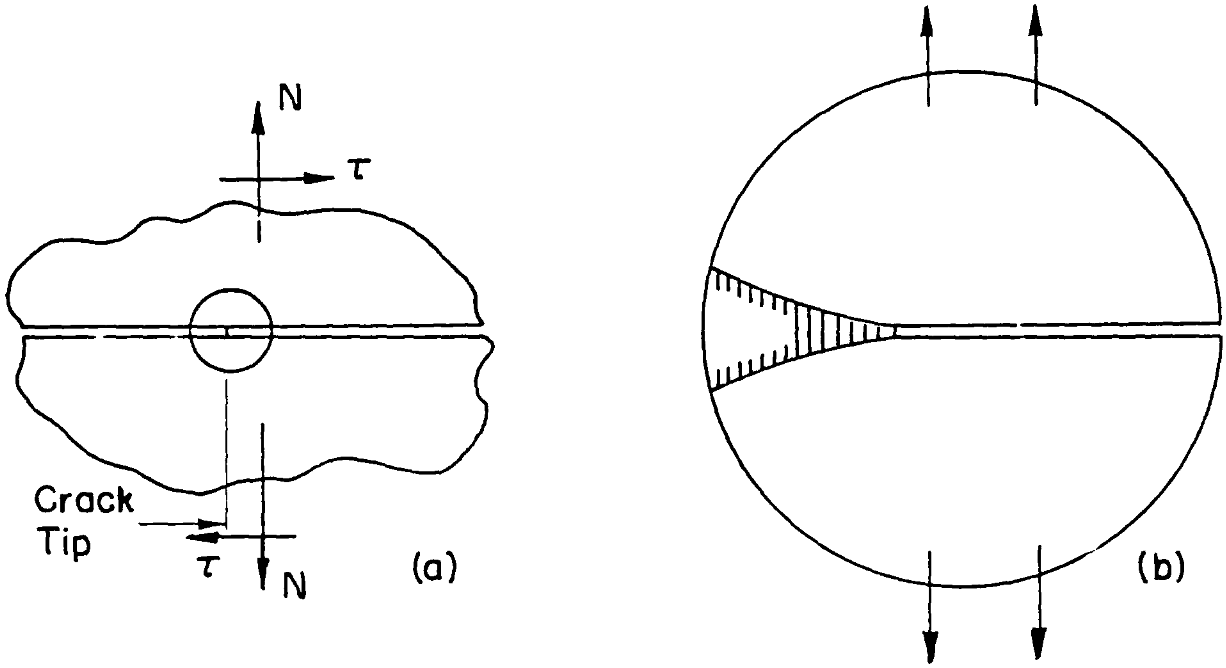

FIG. 1 FAILURE MODEL FOR TOUGH ADHESIVE

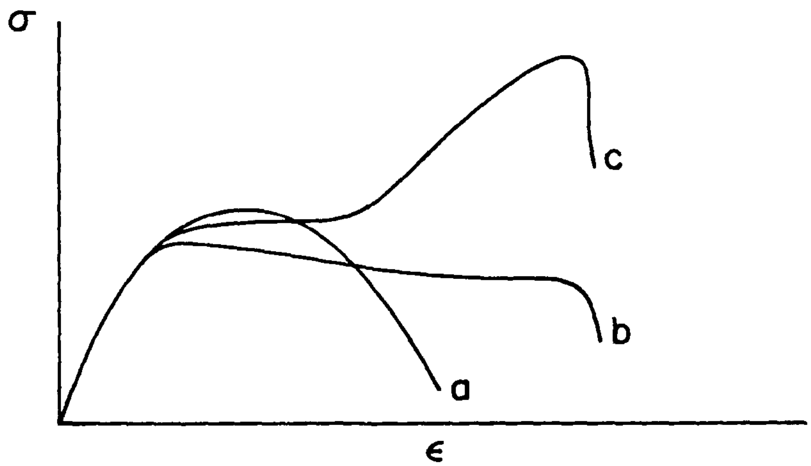

FIG. 2 THREE EXAMPLES OF MATERIAL BEHAVIOUR
LEADING TO CRACK INSTABILITY

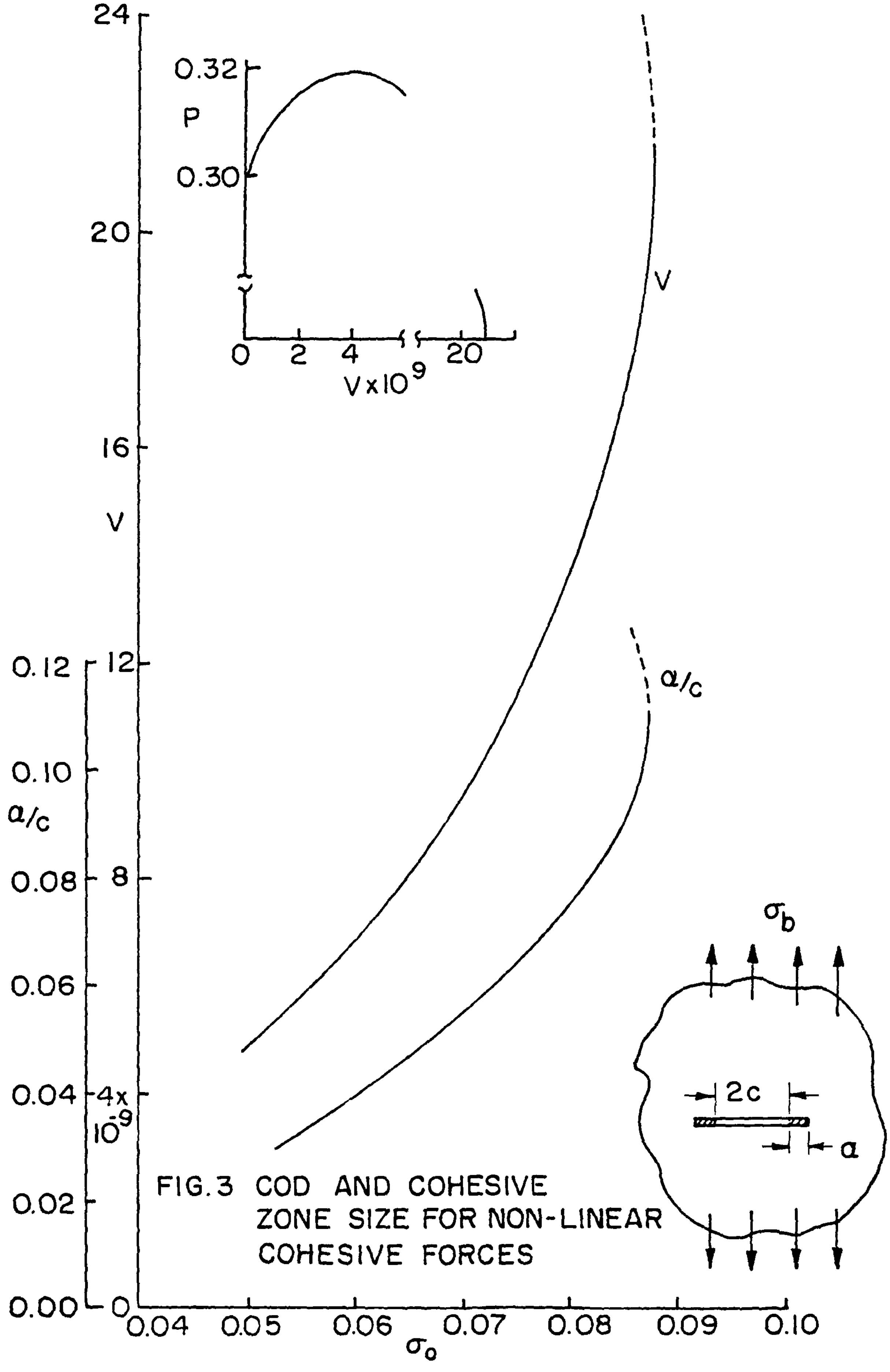

FIG. 3 COD AND COHESIVE ZONE SIZE FOR NON-LINEAR COHESIVE FORCES